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Examination of Suspected Delta II Second-Stage Reentry Debris

15 July 2001

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Prepared for

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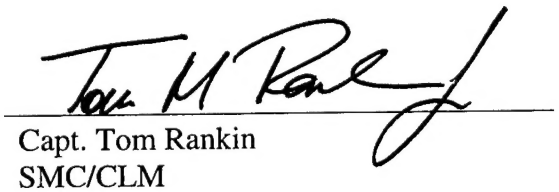

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14. ABSTRACT

A piece of suspected reentry debris was submitted to the Materials Science Department for analysis to determine whether its composition was consistent with its supposed source. The debris consisted of a black-colored woven fabric that locally had clear fused-looking areas, with entrapped bubbles, and small metallic deposits.

The time and location of the recovered debris correspond with the reentry of a Delta II second stage. The second-stage fuel tank, thrust chamber, and a pressure vessel impacted at several locations in Texas, and trajectory analysis, allowing for the prevailing winds, shows that the recovered debris is probably from the reentry event. The chemical composition of the fibers (calcium alumino silicate) and appearance of the piece of debris is consistent with it being from the outer woven E-glass fabric from the Delta second-stage thrust chamber. The metallic deposits on the fabric are believed to be residue from the aluminum flange. E-glass has a "softening" point of about 850°C and a "melting" point of approximately 1200°C. The large number of bubbles trapped in the fused glass is also consistent with the orbital reentry scenario and assumes the material was relatively fluid and had reached a temperature above the melting point of E-glass.

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Ms. Lottie Williams provided the specimen for study and the photographs in Figures 1 and 2. R. Patera provided background information regarding the reentry event. The Aerospace Corporation Flight Mechanics Department performed the trajectory reconstruction and simulated reentry track in Figure 3. I. Lee provided background information and facilitated obtaining the prepreg glass overwrap sample from the manufacturer. The authors would also like to thank The Aerospace Corporation Medium Launch Vehicle and the Air Force SMC/AXF program offices for support of reentry debris recovery and research.

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1. Introduction

A piece of suspected reentry debris was submitted to the Materials Science Department for analysis to determine whether its composition was consistent with its supposed source. The debris was found by Ms. Lottie Williams in Turley, OK (about 7 miles north of Tulsa, OK) at about 4 AM on January 22, 1997. The debris fragment struck her on the shoulder while she was walking in O'Brien Park. She described the debris as being a lightweight metal mesh object about 5 in. in size and sent The Aerospace Corporation Center for Orbital and Reentry Debris Studies (CORDS) a photo and a small portion of the object to analyze (Figures 1 and 2).

The time and location of the recovery of the debris can be correlated with the reentry of a Delta II second stage. The Delta II was launched from Vandenberg Air Force Base (VAFB) on April 24, 1996, carrying the Ballistic Missile Defense Organization (BMDO) Midcourse Space Experiment satellite. The second stage reentered the atmosphere on January 22, 1997. It was predicted that the vehicle broke up at an altitude of 42 nmi over Topeka, KS at 3:36 AM, local time. The second-stage fuel tank, thrust chamber, and a helium sphere were recovered from several sites in Texas.[1] These areas are about 500 mi. south of Turley, OK, which was along the reentry path. Simulation of the reentry track of a light-weight piece of debris with a low ballistic coefficient, taking into account the presumed breakup altitude, reentry track of the other pieces of the Delta second stage, and the prevailing winds, shows that the debris recovered by Ms. Williams could be from the reentry event (Figure 3).

Figure 1. Ms. Lottie Williams holding the debris that struck her.

Figure 2. Photographs of the complete piece of debris (approximately 5 in. in length).

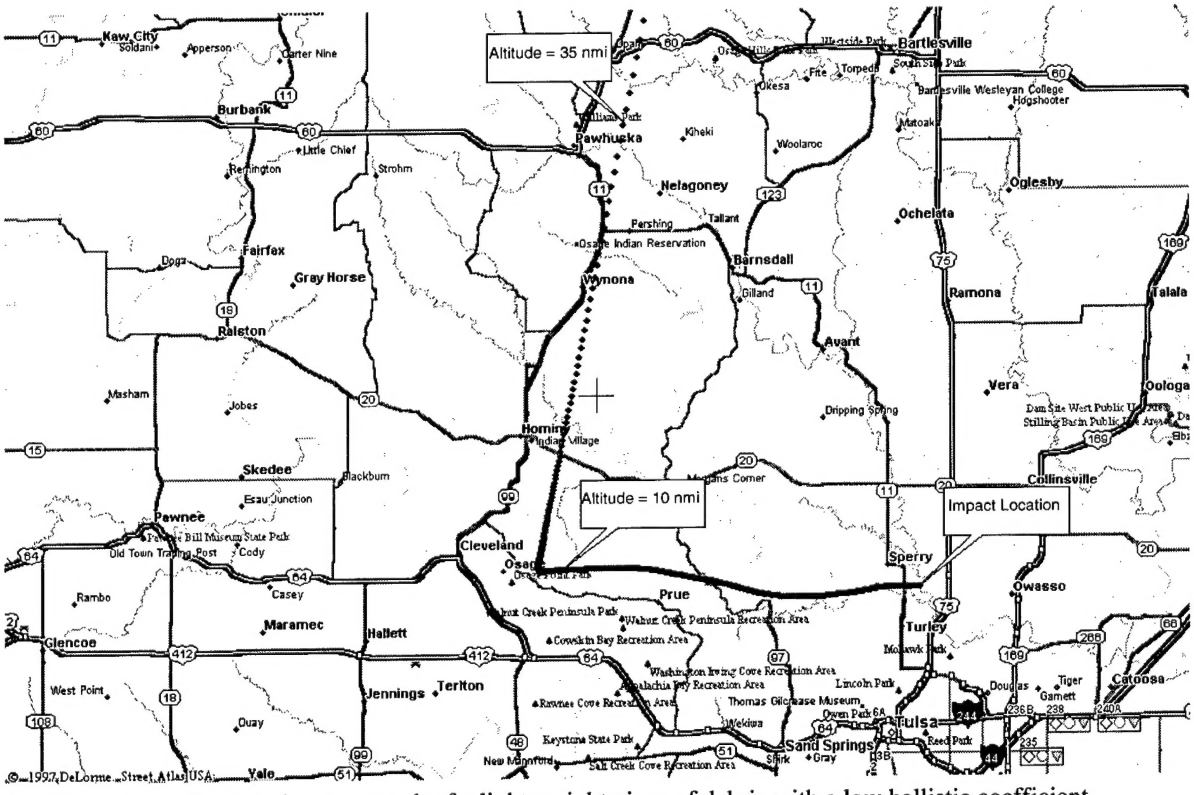

Figure 3. Simulated reentry track of a light-weight piece of debris with a low ballistic coefficient, taking into account the presumed breakup altitude and reentry track of the other pieces of the Delta II second stage.

2. Experimental

The sample was first examined with an optical stereomicroscope, after which small representative pieces of material were removed and coated with a thin layer of carbon in order to make them conductive. These small pieces, along with pieces of the suspected source material were then examined in a JEOL model JSM-840 scanning electron microscope (SEM) equipped with an EDAX Phoenix windowless energy-dispersive X-ray spectrometer (EDXS), which was used to determine the qualitative chemical composition. A small metallic particle in the debris was analyzed by X-ray diffraction (XRD) using nickel-filtered copper radiation and a Gandolfi camera. XRD was used to identify crystalline compounds.

3. Results

Visually the material appeared to be a piece of black-colored woven fabric that was about 1.5 cm x 0.5 cm x 5.0 cm in size (Figure 4). Initially, before closer inspection and handling, it was thought to be a piece of carbon or graphite fiber cloth because of its color; however, it was very hard and brittle. Upon closer examination, it was found that the fibers were colorless, with a very dark coating, and that there were colorless fused-looking deposits that contained many bubbles (Figure 5). There were also a few small areas (1 mm x 1 mm) that appeared silvery-metallic.

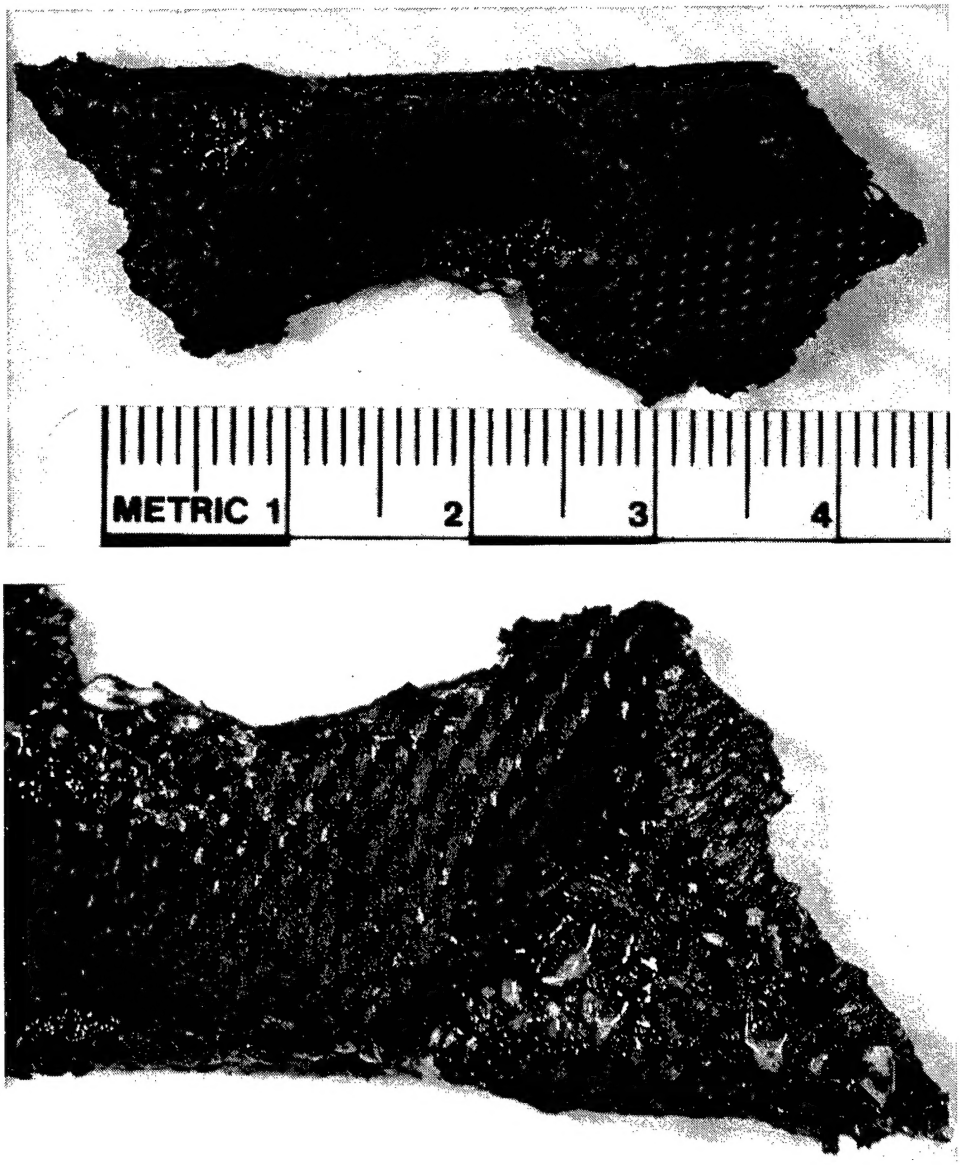

Figure 4. Optical macrophotographs of as-received debris. Upper scale bar is in cm.

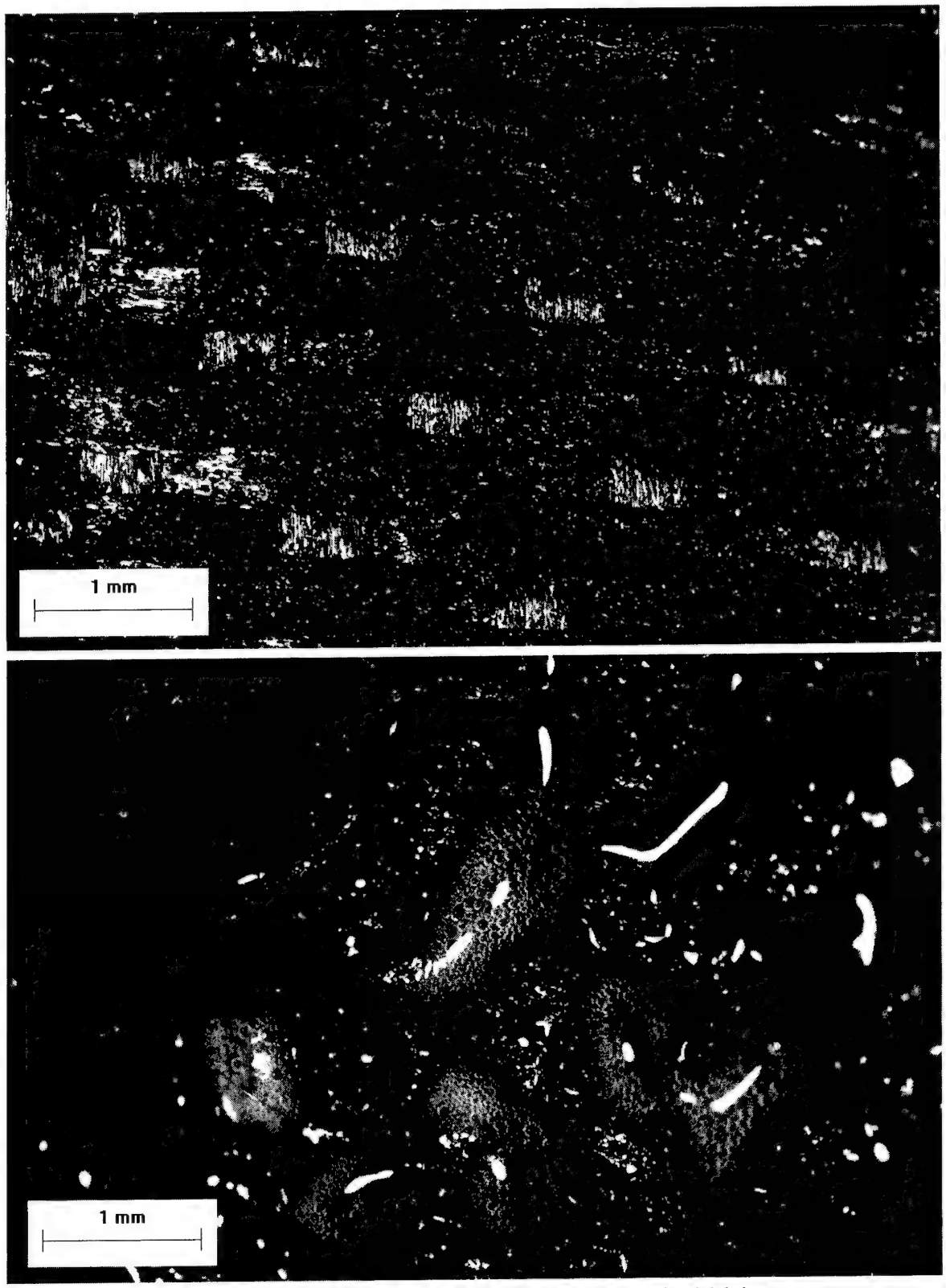

Figure 5. Close-up optical macrophotographs of as-received debris.

In the SEM, the fibers appeared very smooth, circular in cross section (about 9 μm in diameter), and locally covered with thin deposits (Figure 6). The fused-looking areas had smooth surfaces, and in some areas bubbles were observed on fracture surfaces (Figure 7). The chemical analyses of the fibers and fused deposits indicated that they both contained primarily oxygen (O), silicon (Si), calcium (Ca) and aluminum (Al) (Figure 8), which is consistent with the material being fiberglass. It is noted that the EDXS system can not detect lithium or boron. The residue on the fibers (lower photograph in Figure 6) consisted primarily of carbon (C) (Figure 8). The metallic-looking areas had a number of different textures, which ranged from rounded-melted, to folded, to feathery-dendritic (Figures 9 and 10), which probably resulted from the melting and resolidification of the aluminum. Several of the small silvery-metallic deposits were analyzed by EDXS, and all contained primarily aluminum (Al) and oxygen in varying proportions (Figure 11); however, minor amounts of magnesium were occasionally detected. The lower picture in Figure 10 shows the dross layer, which consists of aluminum oxides and impurities, that forms on the surface of molten aluminum that is in contact with an oxidizing atmosphere for a certain length of time. The EDX spectrum from this area did show more oxygen (bottom of Figure 11) than in other metallic areas. The depth of penetration in the EDX analysis is on the order of 1 μm. An XRD pattern of one metallic particle indicated that the bulk of the material was metallic aluminum; however, some very weak lines attributed to alpha aluminum oxide (α-Al_2O_3) were also observed. Based on the XRD and EDX analyses, it is assumed that the aluminum oxide is a thin surface layer.

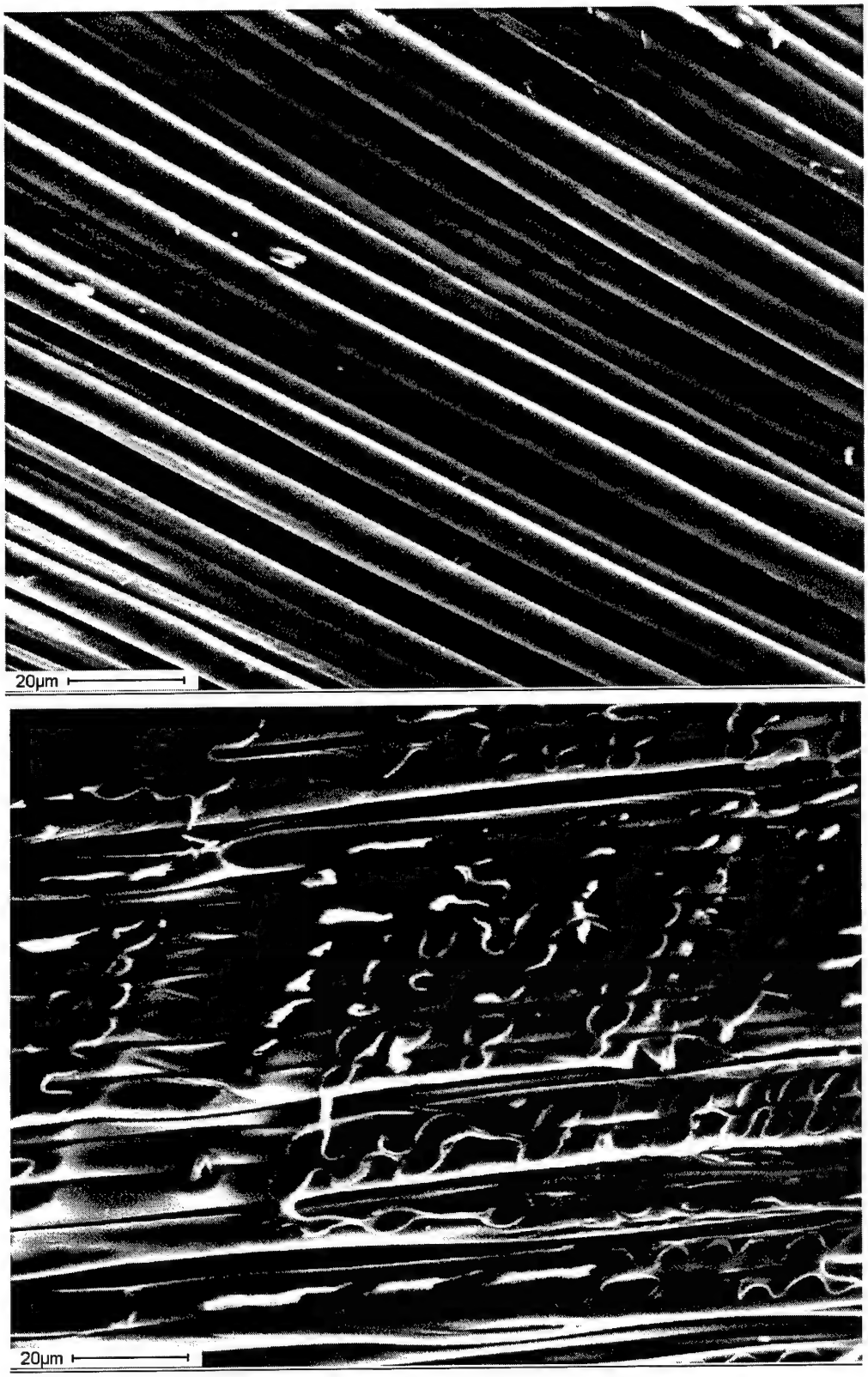

Figure 6. SEM photographs of fibers in the debris.

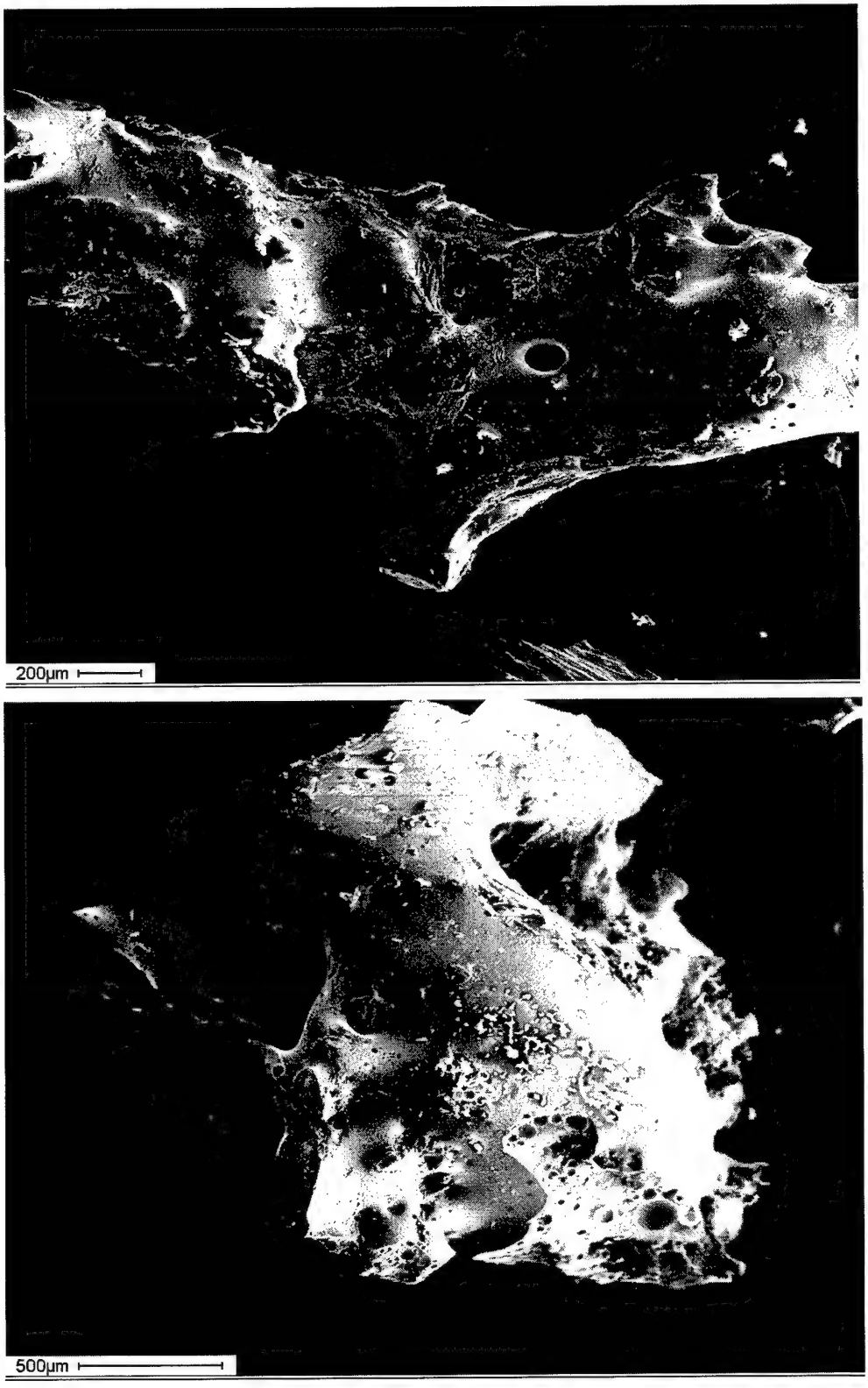

Figure 7. SEM photographs of fused deposits on debris.

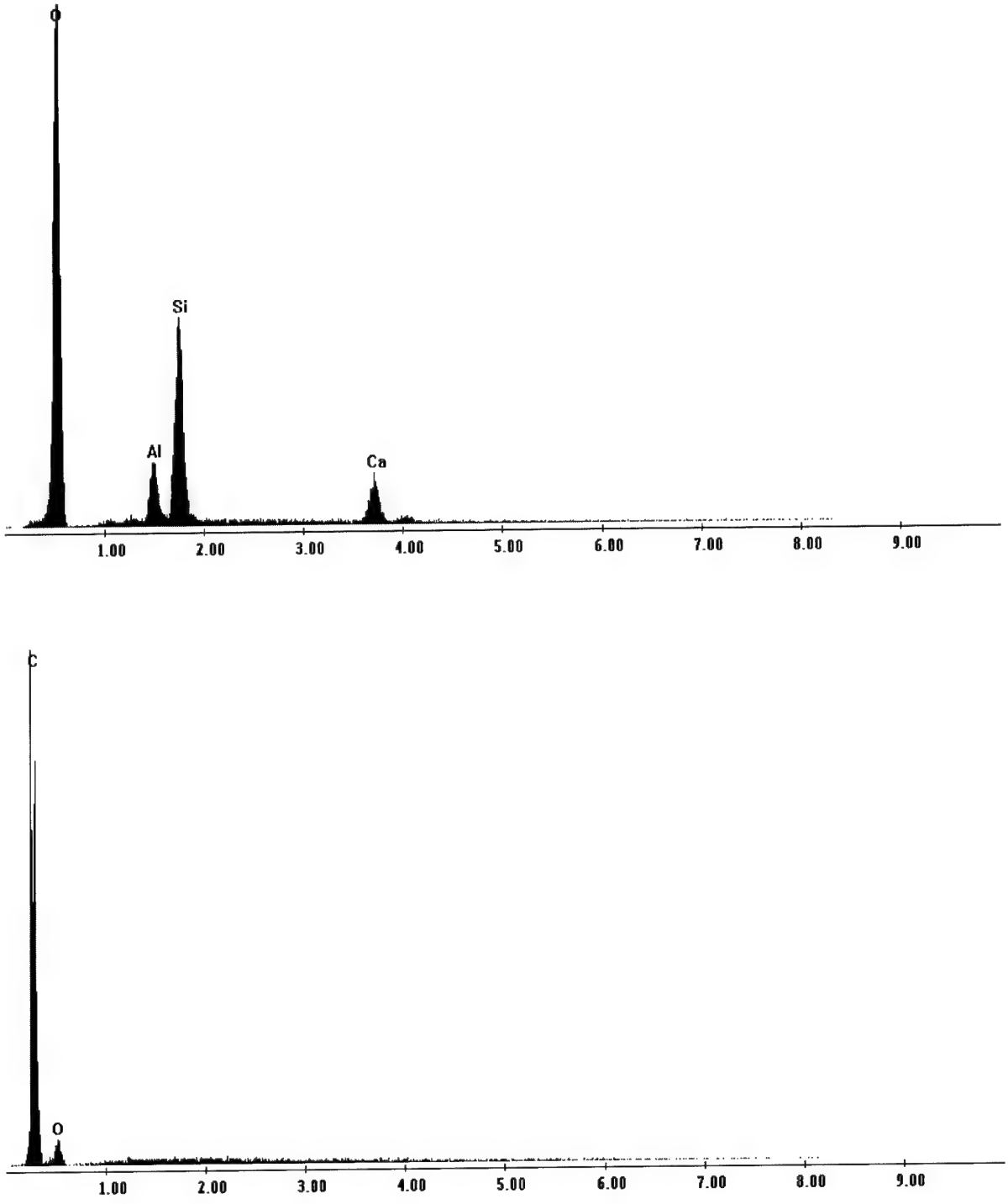

Figure 8. EDX spectra of fibers in debris (top) and deposit on fiber (bottom).

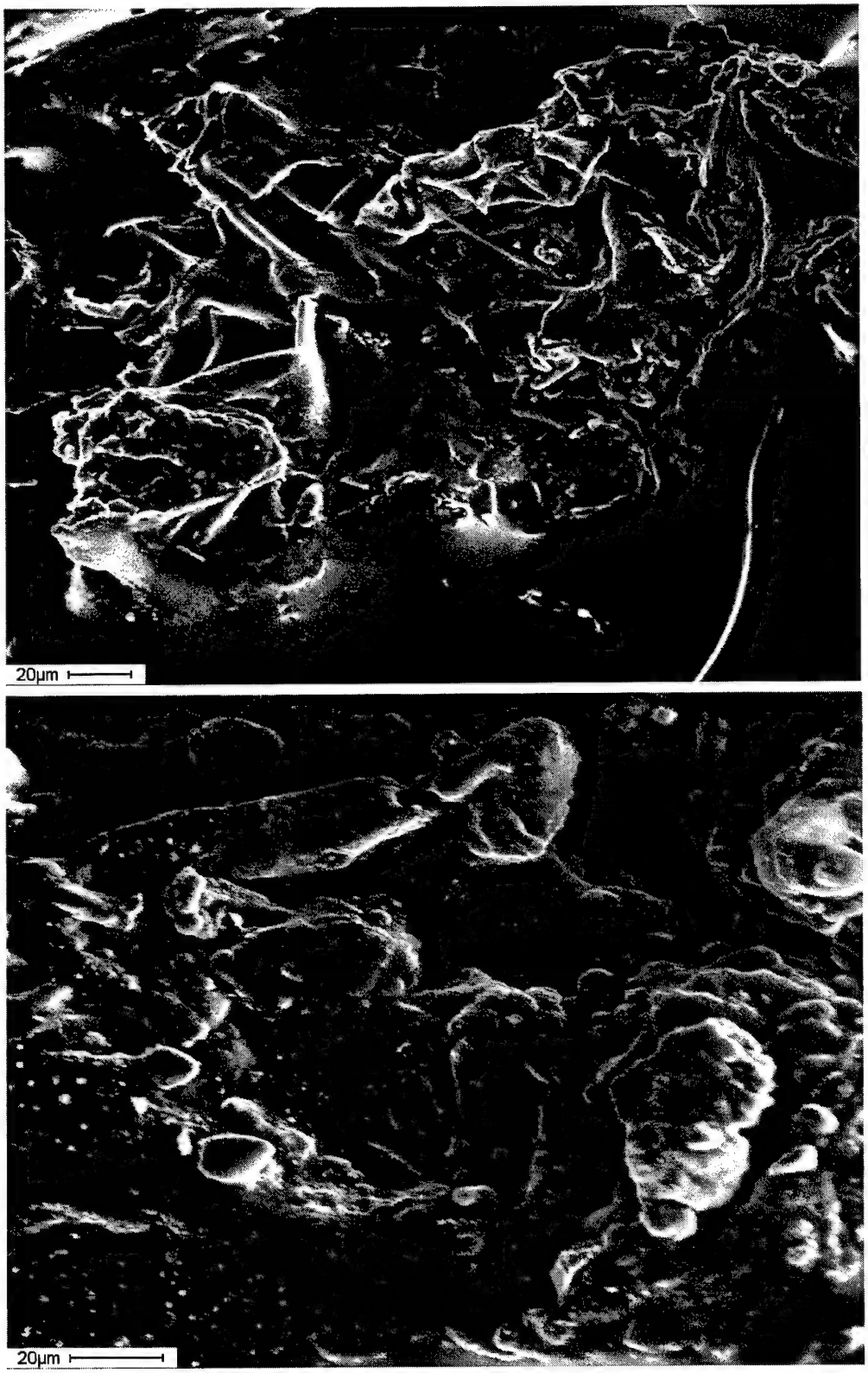

Figure 9. SEM photographs of metallic deposits on debris.

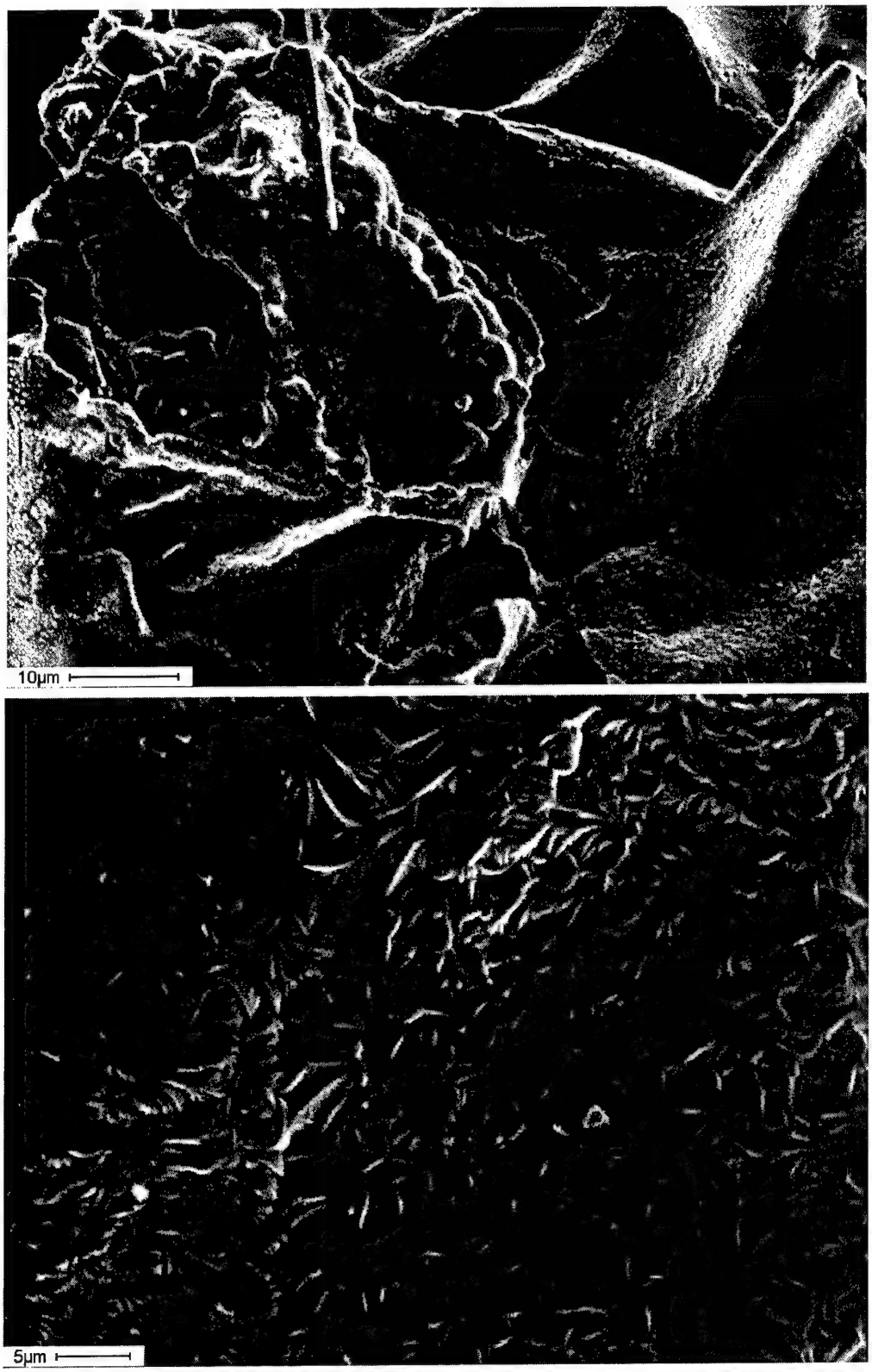

Figure 10. SEM photographs of metallic deposits on debris.

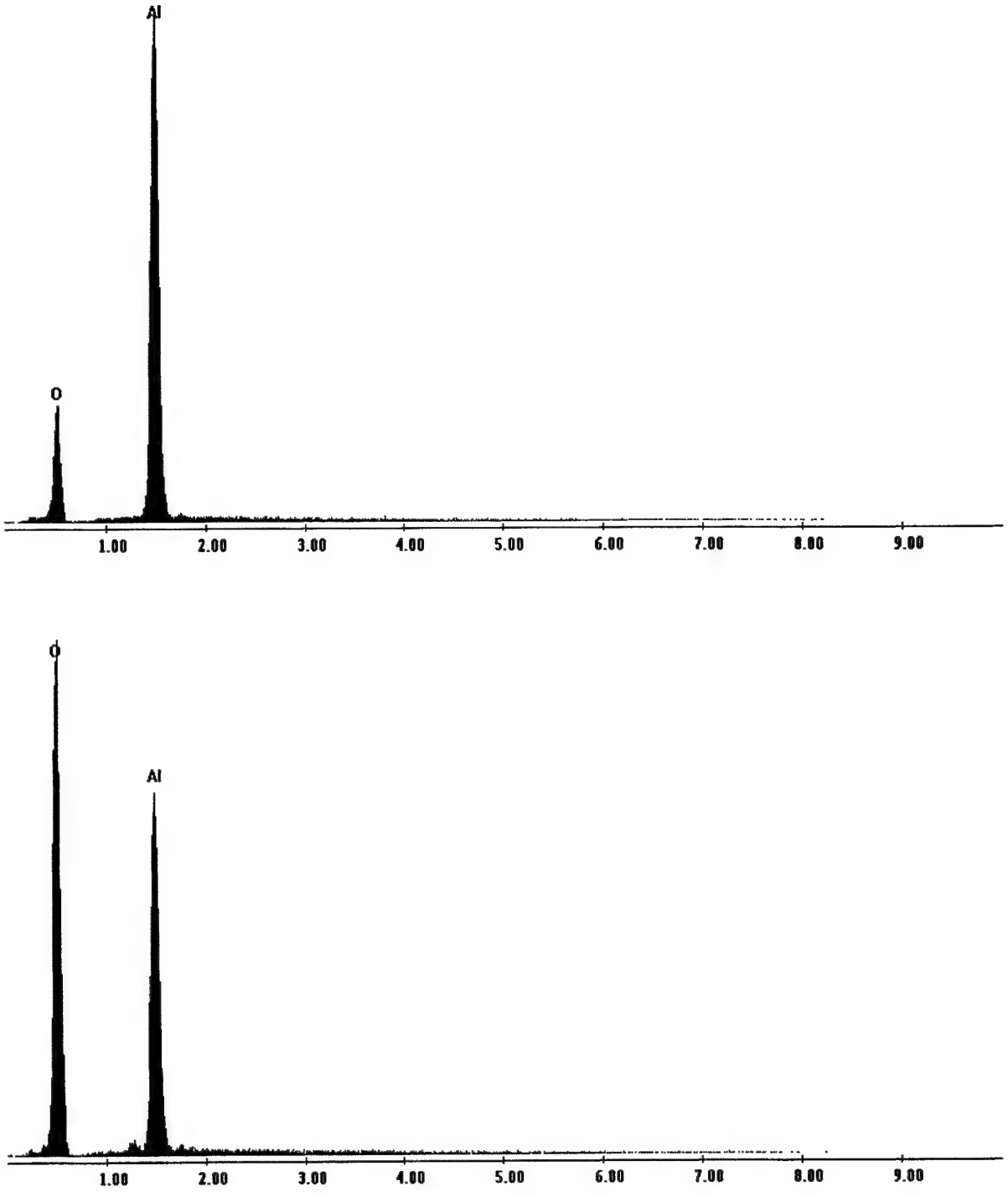

Figure 11. EDX spectra of shiny metallic deposits in debris.

4. Discussion

The chemical composition of the fibers is consistent with their being a type of fiberglass. There are several types of glass fibers, at least one of which (E-glass) has a composition (calcium aluminoborosilicate) consistent with the analyses of the debris (boron can not be detected by EDXS). The fact that the compositions of the fibers and fused deposits were nearly identical implies that the fused material was derived from melting of some of the fibers.

The main body of the Delta II second-stage thrust chamber (referred to as the nozzle in Ref. 1) was recovered from near Seguin, Texas. Upon impact it had broken in the throat region into two pieces. The thrust chamber consists of an inner silica-phenolic liner and an asbestos-phenolic insulator, which is covered with several layers of fiberglass fabric overwrap, in a phenolic matrix, followed by a final layer of glass roving. The forward flange of the thrust chamber is constructed from 6061 aluminum (Al, 1.0% Mg, 0.6% Si, 0.2% Cr, 0.3% Cu), and thin aluminum "fingers" extend aft of the flange. The composition and appearance of the piece of debris are consistent with it being from the outer woven glass fabric from the Delta second-stage thrust chamber. However, the silica-phenolic liner is manufactured from woven fiberglass tape and could be another possible source of the debris. Since the exact compositions of the glass fibers in the silica phenolic liner and glass overwrap were not known, samples of these materials were obtained and examined by SEM/EDXS for comparison with the debris. A piece of the overwrap prepreg (glass fabric with uncured phenolic resin) was obtained from the manufacturer of the thrust chamber. The prepreg was cured at 200°C for one hour in air and subsequently heated in air at 600°C for one hour in order to burn away the phenolic resin and expose the glass fibers. The resulting fabric was white in color. A sectioned portion of a test-fired second-stage thrust chamber was available in one of our labs, and a small portion of the silica-phenolic liner from the ID surface of the chamber was also examined in the SEM. Bundles of woven fibers could be seen visually on the ablated ID surface of the test-fired silica-phenolic liner.

Figures 12 and 13 are SEM photographs of the glass fibers from the silica phenolic and glass fabric overwrap, respectively. The fibers from both materials are similar in size and appearance with the fibers from the debris. Figures 14 and 15 present the EDX spectra of the glass fibers from the silica phenolic and glass fabric overwrap, respectively. It can bee seen that the fibers from the silica phenolic contain only silicon and oxygen, indicating that they were not the source of the debris. However, the spectrum of the overwrap fibers contains oxygen, silicon, calcium, and aluminum, and is nearly identical to that of the debris fibers (Figure 8). Figure 16 compares the debris with the fabric from the overwrap. The weave pattern in each is very similar.

Figure 12. SEM photograph of fibers from silica phenolic liner of test fired thrust chamber.

Figure 13. SEM photograph of fibers from glass fabric overwrap used on thrust chamber.

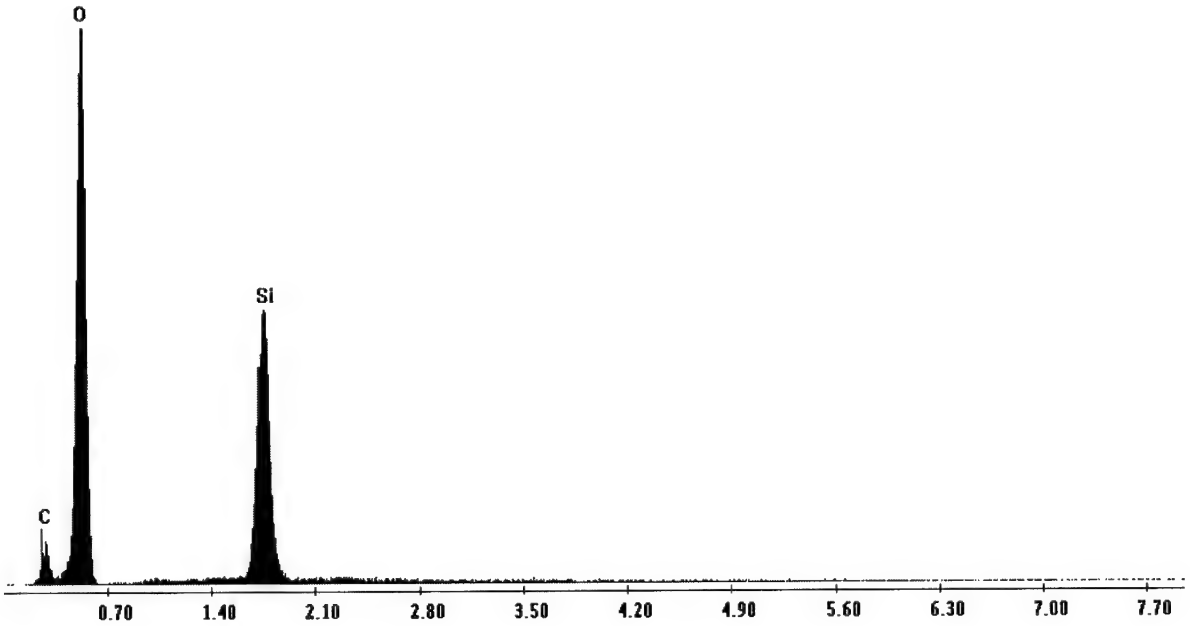

Figure 14. EDX spectrum of fibers from silica phenolic liner.

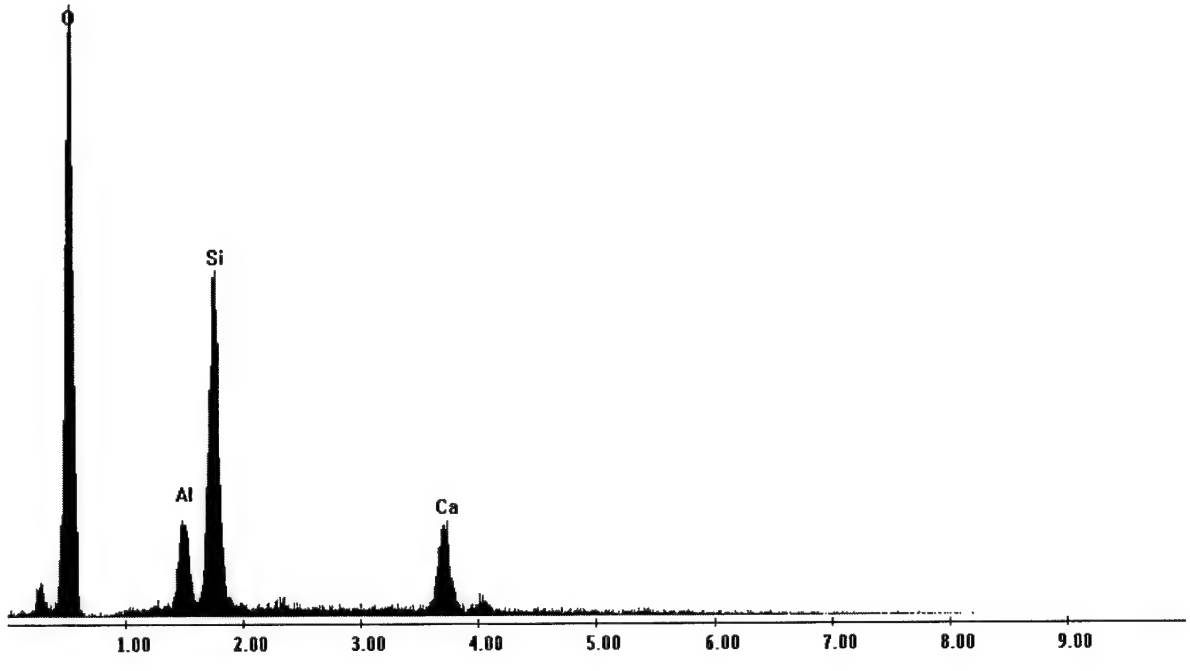

Figure 15. EDX spectrum of fibers from thrust chamber glass fabric overwrap.

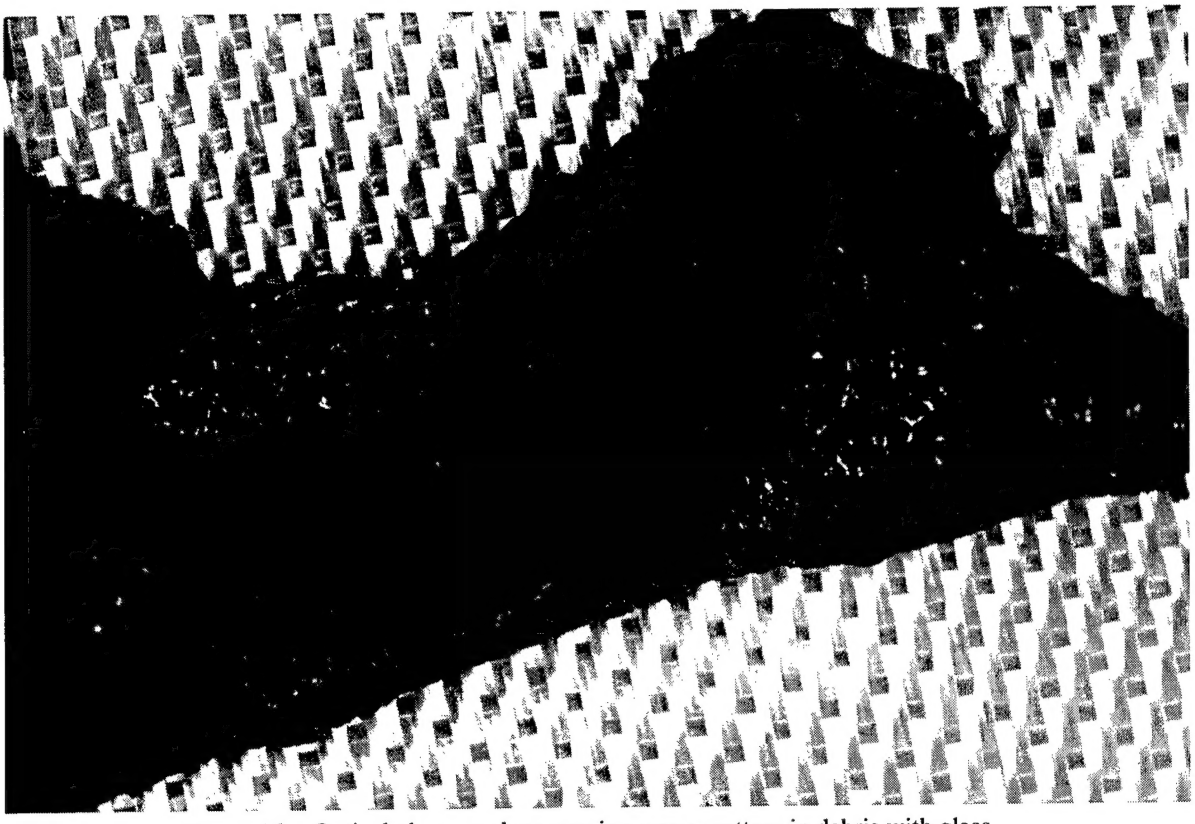

Figure 16. Optical photograph comparing weave pattern in debris with glass overwrap fabric.

5. Summary

The time of the recovery of the debris was consistent with the known reentry of a Delta II second stage based on tracking by the U. S. Space Command and the recovery of verified hardware in Texas. The location where the debris was recovered in Oklahoma is consistent with the reentry of the Delta II second stage based on the second-stage polar orbit, the locations of recovered second-stage components (including the thrust chamber), and a simulated trajectory for the debris from a hypothetical breakup point. The chemical composition and appearance of the piece of debris is consistent with it being from the outer woven glass fabric from the Delta II second-stage thrust chamber. Rapid pyrolysis of the phenolic resin, such as during reentry, probably accounts for the black color of the debris. When phenolic resin is exposed to elevated temperature, in air, at atmospheric pressure, for an extended period of time it completely oxidizes, leaving no residue. Rapid weight loss begins at about 400°C and reaches completion by about 700°C. However, if less oxygen is present, or the heating rate is high and duration is short, it will char, leaving a carbonaceous residue as was observed on the fibers in the debris. The aluminum deposits on the debris fabric are believed to be residue from the thrust chamber forward flange or "fingers." The fact that the aluminum had melted indicates that the reentry temperature exceed 650°C (melting point of 6061 Al). The presence of fused deposits with the same composition as the glass fibers, and containing bubbles, indicates that some of the glass fibers had melted. E-glass has a "softening" point of about 850°C and a "melting" point of approximately 1200°C. The large number of bubbles trapped in the fused glass is also consistent with the orbital reentry scenario and assumes that the material was relatively fluid and had reached a temperature above the melting point of E-glass (1200°C). In comparison, material that might fall from a commercial jetliner, for example, would not be expected to reach these high temperatures.

Reference

1. W. H. Andrus, Jr., "Stage two of Delta II rocket lands in Texas," *MUFON UFO Journal*, March 1997, No. 347, 3–6.

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